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C4X X12  
U1S S1346 S1347 S1387**

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**WO 93/22397 A1  
Chem.Abs. 123:143045 Chem.Abs. 122:33370  
Chem.Abs. 121:302740 Chem.Abs. 121:256718  
Chem.Abs. 120:324302 Chem.Abs. 120:272840  
Chem.Abs. 120:272839 Chem.Abs. 120:165617  
Chem.Abs. 120:19350 Chem.Abs. 119:227845  
Chem.Abs. 119:97676 Chem.Abs. 118:30690  
Chem.Abs. 116:42907 Chem.Abs. 115:115251  
Chem.Abs. 113:192043 Chem.Abs. 113:79530**

(58) Field of Search

**Online: CAS ONLINE**

(54) **Monomeric directive mesogenic compounds, intermediates therefor and polymers therefrom**

(57) The invention relates to directive mesogenic compounds or mixtures thereof comprising mesogen-containing molecule, said mesogens having two side chains attached thereto which contain a terminal polymerizable functional group, said mesogens and said functional groups being separated by at least two to twenty spacer atoms, wherein both spacer groups have different chain length.

The invention is directed to monomeric directive mesogenic compounds as defined in claims 1, 2 and 7; polymers prepared by polymerising such monomers (claim 9); and intermediated (as defined in claim 10) for such monomers.

GB 2 297 549

## Direactive mesogenic compound

The invention relates to

5      direactive mesogenic compounds or mixtures thereof obtainable by

a)      treating a mesogenic diol of formula I,



10

in which

MG      is a mesogenic group.

15      with a mixture of the halides of formula II and III.



20

in which

$\text{X}^1$  and  $\text{X}^2$       are each independently Cl, Br or I

25      m and n      are different integers between 1 and 20

$\text{R}^a$  and  $\text{R}^b$       are each independently groups selected from



30

wherein

W      is H,  $\text{CH}_3$  or Cl.

35      in the presence of a base, and

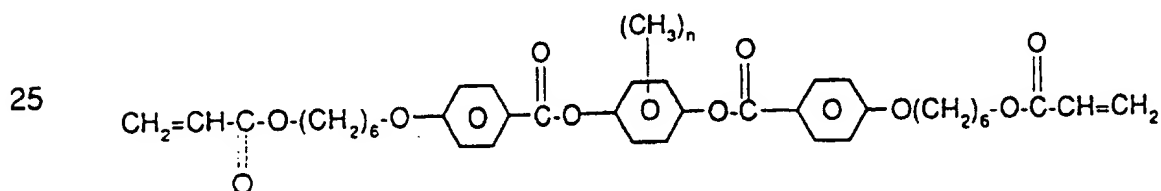
b) treating the resulting intermediate

- in the case of  $R^a$  and  $R^b$  being  $-\text{CH}_2\text{OH}$ , with a vinyl derivative of formula  $\text{CH}_2=\text{CW}-(\text{CO})_a-\text{O}-$  or a reactive derivative thereof, in which  $a$  is 0 or 1
- in the case of  $R^a$  and  $R^b$  being  $-\text{CH}=\text{CWH}$  with a perbenzoic acid.

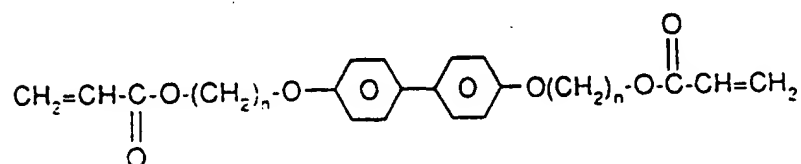
The invention furthermore relates to the preparation of such compounds and to their use in electrooptical scattering systems and for the preparation of oriented liquid crystal polymers.

Reactive liquid crystal compounds can be polymerized in situ, whilst in their liquid crystal phase, to give highly crosslinked anisotropic films which can be used, for example, as polarizing beam splitters (see, for example, EP 0,428,213). Reactive liquid crystal compounds have furthermore been proposed for electrooptical scattering systems (see, for example, EP 0,451,905), cholesteric polarizers (e.g. EP 0,606,940) and compensation films for STN displays (e.g. EP 0,423,881).

Reactive liquid crystal diesters of formula

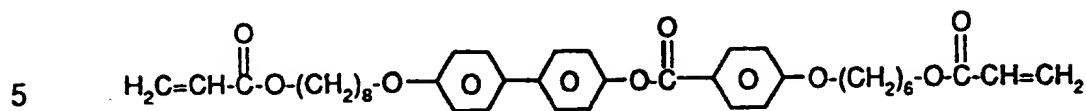


are mentioned in EP 0,261,712 ( $n=0$ ), EP 0,331,233 ( $n=1$ ). Reactive liquid crystal biphenyls of formula



are disclosed by EP 0,405,713.

The International Patent application WO 93/22397 discloses a compound of formula



10 These reactive liquid crystalline compounds often exhibit, however, rather high melting points disadvantageous values of the birefringence and comparable narrow mesophase ranges.

In view of the broad range of applications of reactive liquid crystal compounds it was desirable to have available further compounds of this type which fulfill the various requirements such as a reasonably low melting point, a high birefringence, a broad mesogenic range and preferably an enantiotropic nematic range to a high degree.

20 It was an object of the present invention to provide new reactive liquid crystalline compounds with advantageous properties thus extending the pool of reactive liquid crystal compounds available to the expert. Other objects of the present invention can be taken from the following detailed specification.

25 The present invention thus relates to reactive mesogenic compounds or mixtures thereof obtainable by treating mesogenic diols of formula I, in particular those having a symmetric structure unit with a mixture of halides of formula II and III and to their use in electrooptical systems of scattering type and for the preparation of oriented liquid crystal polymers. The invention furthermore relates to the preparation of compounds according to formula I.

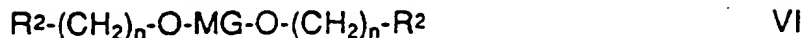
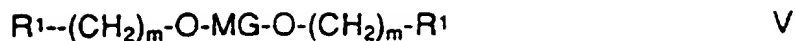
30 Preferred embodiments of the present invention are:

- a) Composition of direactive compounds comprising at least one compound of each formula IV, V and VI,

35

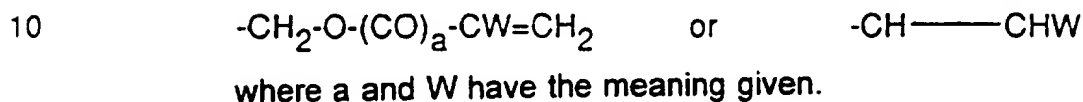


IV



5 in which MG, m and n have the meaning given, and

$R^1$  and  $R^2$  are each independently



b) Direactive compound or mixture thereof

15 in which

$m - n \geq 1$ , in particular 2, 3 or 4.

c) Direactive compound or mixture thereof in which

20 MG is a mesogenic group of formula VII,



25 in which

$A^1$  and  $A^2$  are each independently

30 (a) 1,4-phenylene in which one or two CH groups may be replaced by N;

(b) 1,4-cyclohexylene in which one or two non-adjacent  $CH_2$  groups may be replaced by -O- or one -CH- group may be replaced by -C(CN)-;

35

(c) naphthalene-2,6-diyl;

it being possible that group (a) is substituted by halogen  
cyano or alkyl, alkoxy or alkanoyl with 1 to 6 C atoms,

5

Z<sup>1</sup>

is each independently -COO-, -O-CO-, -CH<sub>2</sub>-CH<sub>2</sub>-,  
-C≡C-, -CH<sub>2</sub>O-, -OCH<sub>2</sub>- or a single bond, and

o

is 1, 2 or 3.

10

15

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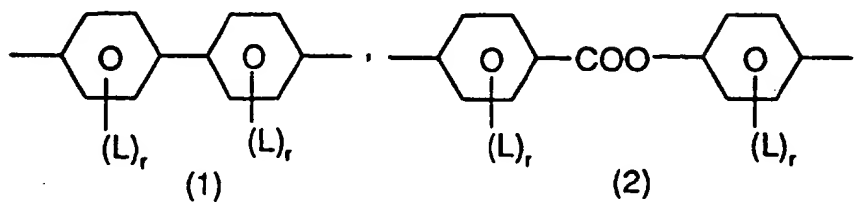
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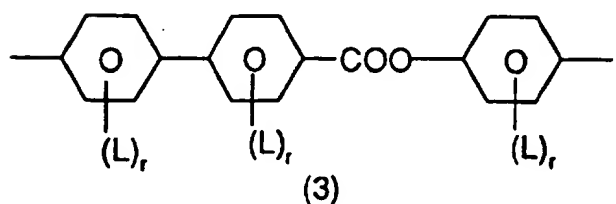
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- d) Direactive compound or mixture thereof in which MG is selected from the structure elements (1) to (6).

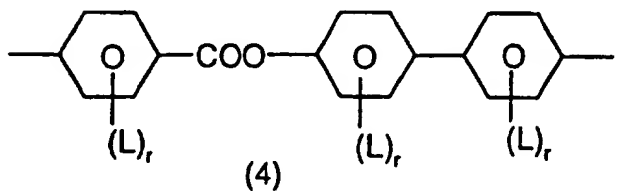
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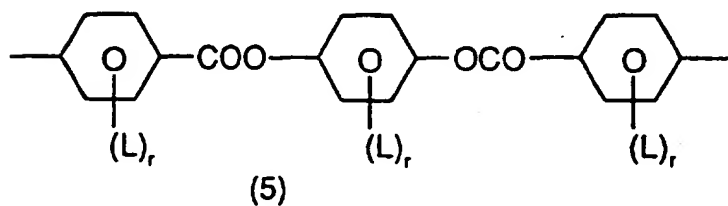
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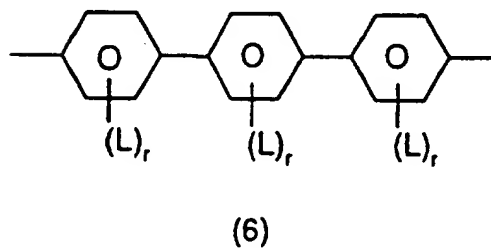
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20



25



in which

30

L is CH<sub>3</sub>, Cl, F, OCH<sub>3</sub> or -CO-CH<sub>3</sub>, and

r is 0, 1, 2 or 4.

35

e) Direactive compound or mixture thereof in which

n and m are given by the following table:

5	m	5	5	5	4	4
	n	2	3	4	2	3

A further aspect of the present invention are direactive compounds of formula IV A



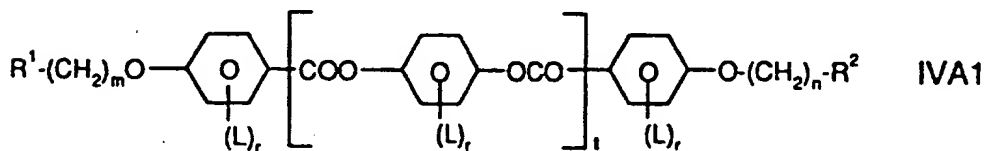
in which

R<sup>1</sup> and R<sup>2</sup> have the meaning given,

m and n are different integers between 2 and 10, and

MG is a mesogenic group, the core of which being symmetrical, preferably a structure element of formula (1), (5) or (6), in particular

direactive compounds of the formula IVA1



in which

R<sup>1</sup>, R<sup>2</sup>,  
L and r have the meaning given,



m and n are different integers between 2 and 10, and

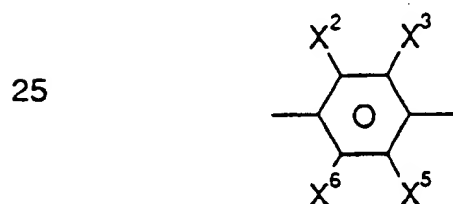
t is 0 or 1.

5 Another aspect of the invention are the polymers prepared by polymerizing a monomer according to any of the preceding claims and

10 chemical intermediate compounds or mixtures thereof useful in preparing direactive compounds or mixtures thereof according to any of the Claim 1 to 8, comprising mesogen-containing molecules, said mesogens having two side chains attached thereto which contain hydroxyl or vinyl group at the end thereof, said mesogens and said functional groups being separated by at least two to twenty spacer atoms, wherein both spacer groups have different chain length.

15 Above and below, the term reactive mesogenic compounds refers to reactive rod-like molecules which may be enantiotropic, monotropic or isotropic, preferably, however, enantiotropic or monotropic.

20 In the inventive compounds in which MG is a mesogenic group of formula VII, A<sup>1</sup> and A<sup>2</sup> can be independently from each other an unsubstituted or a substituted 1,4-phenylene group of formula



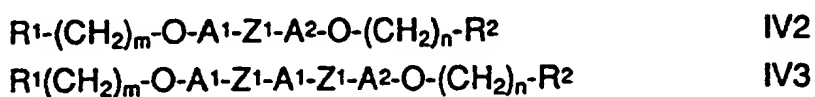
30 X<sub>2</sub>, X<sub>3</sub>, X<sub>5</sub> and X<sub>6</sub> can be independently from each other H, F, Cl, methyl or CN.

In the following, for the sake of simplicity, the following notation will be used:

35

Phe. 2 X<sup>2</sup> 3 X<sup>3</sup> 5 X<sup>5</sup> 6 X<sup>6</sup> is a 1,4-phenylene group carrying in 2-position the group X<sup>2</sup>, in 3-position the group X<sup>3</sup> etc.; in case X<sup>2</sup>, X<sup>3</sup>, X<sup>5</sup> and/or X<sup>6</sup>, denote H, this will not be specified in above notation, i.e. only true substitutions will be listed. Thus Phe, for example, is an unsubstituted 1,4-phenylene group while Phe.2F 5 Cl is a 2-fluoro-5-chloro-1,4-phenylene group. Furthermore, Pyr is pyrimidine-2,5-diyl, Pyd is pyridine -2,5-diyl and Nap is a naphthalene-2,6-diyl group. The notation Pyr and Pyd in each case include the 2 possible positional isomers.

The compounds according to formula IV comprise 2- and 3-ring compounds (n=1 or 2) of formula IV2 and IV3:



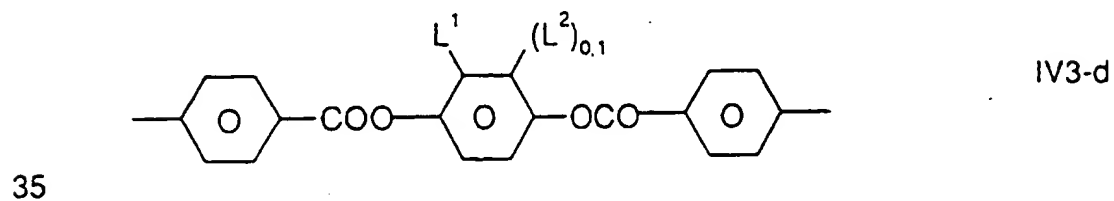
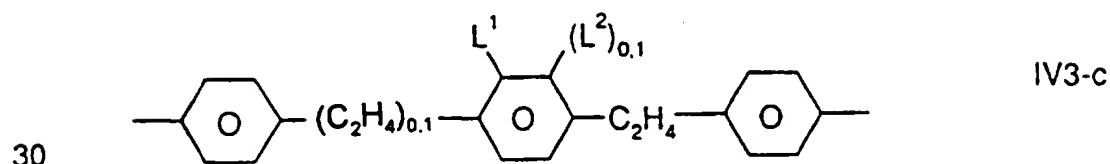
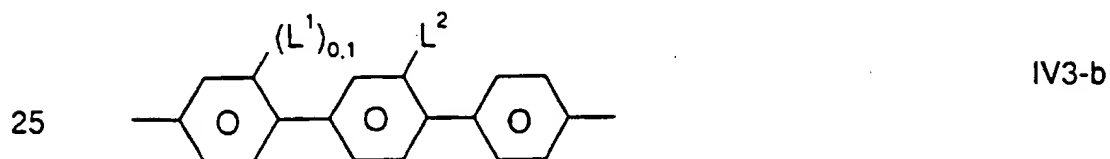
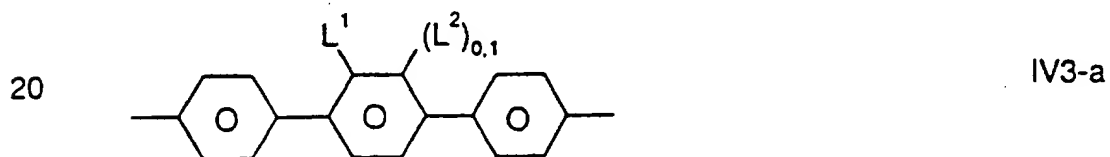
In the 3-ring compounds of formula IV3, the ring groups A<sup>1</sup> can be chosen independently from each other.

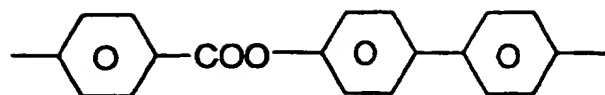
Especially preferred is a smaller group of 2-ring compounds exhibiting the following structures for -A<sup>1</sup>-Z<sup>1</sup>-A<sup>2</sup>-:

-Phe.2CH <sub>3</sub> -Phe-	IV2-1
-Phe.3CH <sub>3</sub> -Phe-	IV2-2
-Phe.2Cl-Phe-	IV2-3
-Phe.3Cl-Phe-	IV2-4
-Phe.2CN-Phe-	IV2-5
-Phe.3CN-Phe-	IV2-6
-Phe.2Cl3Cl-Phe-	IV2-7
-Phe.2Cl3F-Phe-	IV2-8
-Phe.2F-Phe-	IV2-9
-Phe.3F-Phe-	IV2-10
-Phe.-Phe-	IV2-11
-Phe.F-Nap-	IV2-12
-Phe.2Cl-Nap-	IV2-13

	-Phe.F-Nap-	IV2-14
	-Phe.3Cl-Nap-	IV2-15
	-Phe.2F-Pyr-	IV2-16
	-Phe.2F-Pyr-	IV2-17
5	-Phe.2CH <sub>3</sub> -Pyd-	IV2-18
	-Phe.2Cl-Pyd-	IV2-19
	-Phe.F-CH <sub>2</sub> CH <sub>2</sub> -Phe-	IV2-20
	-Phe.3F-CH <sub>2</sub> CH <sub>2</sub> -Phe-	IV2-21
	-Phe.2Cl-CH <sub>2</sub> CH <sub>2</sub> -Phe-	IV2-22
10	-Phe.3Cl-CH <sub>2</sub> CH <sub>2</sub> -Phe-	IV2-23
	-Phe.2CN-CH <sub>2</sub> CH <sub>2</sub> -Phe-	IV2-24
	-Phe.3CN-CH <sub>2</sub> CH <sub>2</sub> -Phe-	IV2-25
	-Phe.2Cl3Cl-CH <sub>2</sub> CH <sub>2</sub> -Phe-	IV2-26
	-Phe.2Cl3F-CH <sub>2</sub> CH <sub>2</sub> -Phe-	IV2-27
15		

The 3-ring compounds according to formula IV3 preferably exhibit the following structures for -A<sup>1</sup>-Z<sup>1</sup>-A<sup>1</sup>-Z<sup>1</sup>-A<sup>2</sup>:





IV3-e

5 In these structures, IV3-a to IV3-d, L<sup>1</sup> and L<sup>2</sup> denote independently from each other H, -Cl, -F, -CN and C<sub>1</sub>H<sub>2+1-5</sub> and, in particular, -Cl, -F, -CN, -CH<sub>3</sub>, and/or -C<sub>2</sub>H<sub>5</sub>.

10 Especially preferred are the following patterns:

	-Phe-Phe.2CH <sub>3</sub> -Phe-	IV3-a-1
	-Phe-Phe.2Cl-Phe-	IV3-a-2
	-Phe-Phe.2CN-Phe-	IV3-a-3
15	-Phe-Phe.2F-Phe-	IV3-a-4
	-Phe-Phe-Phe-	IV3-a-5
	-Phe-Phe.2C <sub>2</sub> H <sub>5</sub> -Phe-	IV3-a-6
	-Phe-Phe.3CH <sub>3</sub> -Phe-	IV3-b-1
20	-Phe-Phe.3Cl-Phe-	IV3-b-2
	-Phe-Phe.3CN-Phe-	IV3-b-3
	-Phe-Phe.3F-Phe-	IV3-b-4
	-Phe-Phe.3C <sub>2</sub> H <sub>5</sub> -Phe-	IV3-b-5
	-Phe.3F-Phe.3Cl-Phe-	IV3-b-6
25	-Phe.3F-Phe.3CH <sub>3</sub> -Phe-	IV3-b-7
	-Phe.3Cl-Phe.3Cl-Phe-	IV3-b-8
	-Phe.3Cl-Phe.3CH <sub>3</sub> -Phe-	IV3-b-9
	-Phe-Phe.2Cl-Phe.3Cl-	IV3-b-10
	-Phe-Phe.3Cl-Phe.3Cl-	IV3-b-11
30	-Phe-Phe.2Cl-Phe.2Cl-	IV3-b-12
	-Phe-Phe.3Cl-Phe.2Cl-	IV3-b-13
	-Phe-Phe.2CH <sub>3</sub> -Phe.3Cl-	IV3-b-14
	-Phe-Phe.3CH <sub>3</sub> -Phe.3Cl-	IV3-b-15
35	-Phe-Phe.2CH <sub>3</sub> -Phe.2Cl-	IV3-b-16

	-Phe-Phe.3CH <sub>3</sub> -Phe.2Cl-	IV3-b-17
	-Phe-Phe.2F-Phe.3Cl-	IV3-b-18
	-Phe-Phe.3F-Phe.3Cl-	IV3-b-19
	-Phe-Phe.2F-Phe.2Cl-	IV3-b-20
5	-Phe-Phe.3F-Phe.2Cl-	IV3-b-21
	-Phe-Phe.2Cl-Phe.3CN-	IV3-b-22
	-Phe-Phe.3Cl-Phe.3CN-	IV3-b-23
	-Phe-Phe.2Cl-Phe.2CN-	IV3-b-24
	-Phe-Phe.3Cl-Phe.2CN-	IV3-b-25
10	-Phe-Phe.2CH <sub>3</sub> -Phe.3CN-	IV3-b-26
	-Phe-Phe.3CH <sub>3</sub> -Phe.3CN-	IV3-b-27
	-Phe-Phe.2CH <sub>3</sub> -Phe.2CN-	IV3-b-28
	-Phe-Phe.3CH <sub>3</sub> -Phe.2CN-	IV3-b-29
	-Phe-Phe.3F-Phe.3CN-	IV3-b-30
15	-Phe-Phe.2F-Phe.3CN-	IV3-b-31
	-Phe-Phe.3F-Phe.2CN-	IV3-b-32
	-Phe-Phe.2F-Phe.2CN-	IV3-b-33
	-Phe-Phe.2F-Phe.2F-	IV3-b-34
	-Phe-Phe.3F-Phe.3F-	IV3-b-35
20		
	-Phe-Phe.2CH <sub>3</sub> -C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-1
	-Phe-Phe.2Cl-C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-2
	-Phe-Phe.2CN-C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-3
	-Phe-Phe.2F-C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-4
25	-Phe-Phe.2C <sub>2</sub> H <sub>5</sub> -C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-5
	-Phe-Phe.2Cl3F-C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-6
	-Phe-Phe.2Cl3Cl-C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-7
	-Phe-C <sub>2</sub> H <sub>4</sub> -Phe.2CH <sub>3</sub> -C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-8
	-Phe-C <sub>2</sub> H <sub>4</sub> -Phe.2Cl-C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-9
30	-Phe-C <sub>2</sub> H <sub>4</sub> -Phe.2CN-C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-10
	-Phe-C <sub>2</sub> H <sub>4</sub> -Phe.F-C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-11
	-Phe-C <sub>2</sub> H <sub>4</sub> -Phe.2OCF <sub>3</sub> -C <sub>2</sub> H <sub>4</sub> -Phe-	IV3-c-12
	-Phe-COO-Phe.OCO-Phe-	IV3-d-1
35	-Phe-COO-Phe.2CH <sub>3</sub> -OCO-Phe-	IV3-d-2

	-Phe-COO-Phe.3CH <sub>3</sub> -OCO-Phe-	IV3-d-3
	-Phe-COO-Phe.2CH <sub>3</sub> 3CH <sub>3</sub> -OCO-Phe-	IV3-d-4
	-Phe-COO-Phe.2OCH <sub>3</sub> -OCO-Phe-	IV3-d-5
	-Phe-COO-Phe.2Cl-OCO-Phe-	IV3-d-6
5	-Phe-COO-Phe.2F-OCO-Phe-	IV3-d-7
	-Phe-COO-Phe.2F3F-OCO-Phe-	IV3-d-8
	-Phe-COO-PhePhe-	IV3-e-1
	-Phe-COO-Phe.2FPhe-	IV3-e-2
10	-Phe-COO-Phe.3FPhe-	IV3-e-3
	-Phe-COO-PhePhe.2F-	IV3-e-4
	-Phe-COO-PhePhe.3F-	IV3-e-5
	-Phe.2F-COO-PhePhe-	IV3-e-6
	-Phe.3F-COO-PhePhe-	IV3-e-7

15


It was observed that the stability of 3-ring compounds wherein one of the 2 groups Z<sup>1</sup> is -COO- or -OCO- while the other denotes a single bond, can be increased if the compound is laterally di- or higher substituted, particularly di-substituted by -Cl, -F, -CN and/or -CH<sub>3</sub>. Compounds of this type are preferred.

20

Especially preferred are further 3-ring compounds where both groups Z<sup>1</sup> are either -COO-, or -OCO- and at least one of the rings A<sup>1</sup>, A<sup>1'</sup> and A<sup>2</sup> are at least mono substituted.

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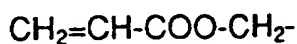
In the compounds of formula IV R<sup>1</sup> is CH<sub>2</sub> = CW-COO-CH<sub>2</sub>-, CH<sub>2</sub> =

CH-O-CH<sub>2</sub>-,  HW-C-C-, with W being H, Cl or alkyl with 1-5 C atoms and m being 1-7.

30

Preferably, R<sup>1</sup> and R<sup>2</sup> is a vinyl group, an acrylate group, an epoxy group and especially preferred are the following means of R<sup>1</sup> and R<sup>2</sup>:

35



R<sup>1</sup>-1

	$\begin{array}{c} \text{CH}_2=\text{C}-\text{COO}-\text{CH}_2- \\   \\ \text{CH}_3 \end{array}$	R <sup>1</sup> -2
5	$\begin{array}{c} \text{CH}_2=\text{C}-\text{COO}-\text{CH}_2- \\   \\ \text{Cl} \end{array}$	R <sup>1</sup> -3
	$\text{CH}_2=\text{CH}-\text{O}-\text{CH}_2-$	R <sup>1</sup> -4
10	$\begin{array}{c} \text{O} \\ \diagup \quad \diagdown \\ \text{H}_2-\text{C}-\text{C}- \end{array}$	R <sup>1</sup> -5

with alkyl denoting C<sub>1</sub>-C<sub>3</sub>-alkyl and m being 1-5.

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The reaction methods mentioned are briefly summarized in the following synthetic tree:

Scheme I

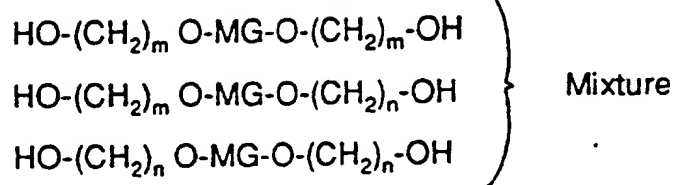
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+  
Mixture of  
 $\text{Br}-(\text{CH}_2)_m-\text{OH}$   
 $\text{Br}-(\text{CH}_2)_n-\text{OH}$   
Butanone,  $\text{K}_2\text{CO}_3$ , heat

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Butylvinyl ether  
DCM  
1,10-Phenanthroline  
Pd (II)-Acetate



Mixture of  
"diacrylates"

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Mixture of  
"Divinylethers"

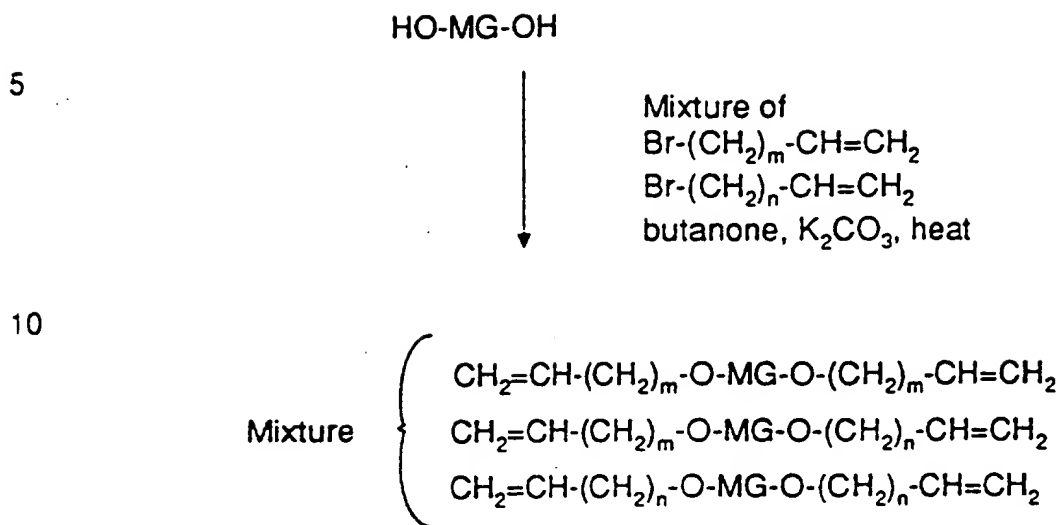
DCM = dichloromethane

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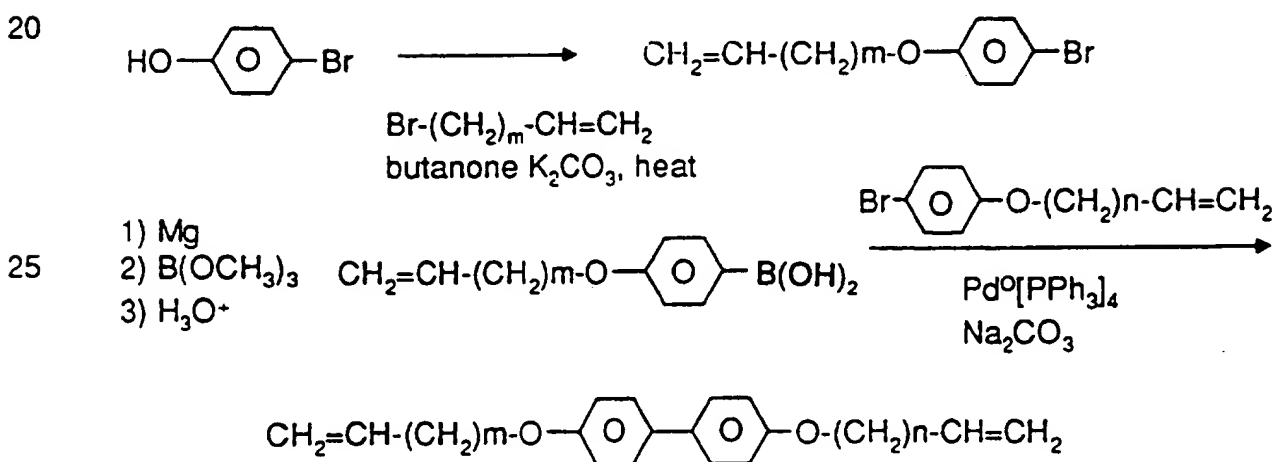
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Scheme II

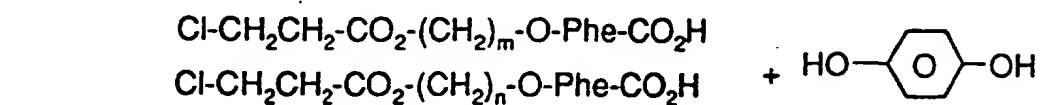


Scheme III

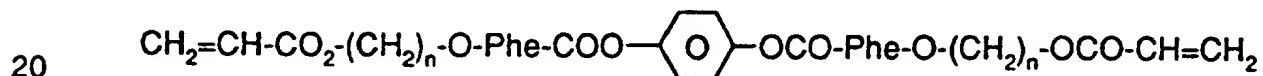
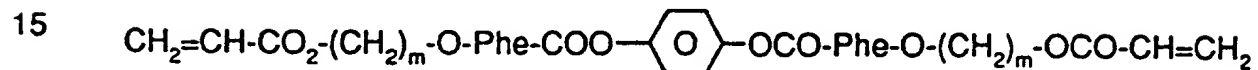


Certain compounds or mixtures according to the invention are obtainable in a "one-pot-synthesis" as outlined in scheme IV:

5 Scheme IV



1) TFAA/DCM  
2) TEA/DCM



TFAA = trifluoroacetic acid  
TEA = triethylamine

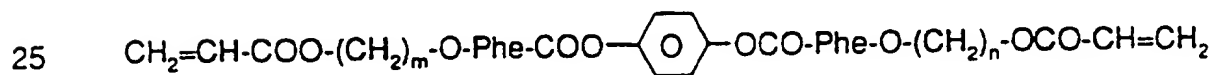
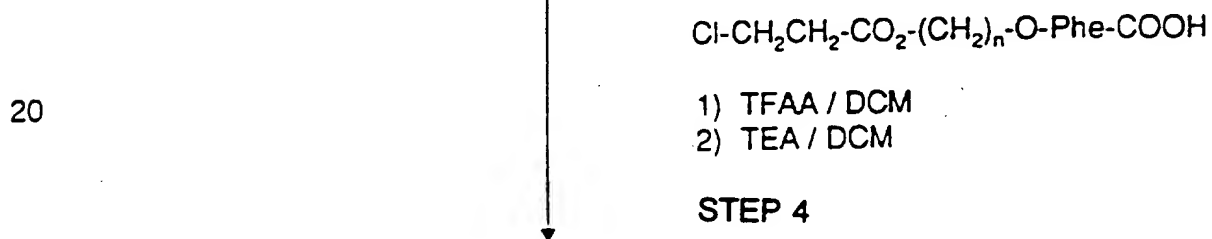
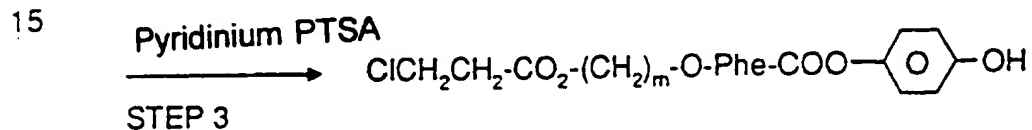
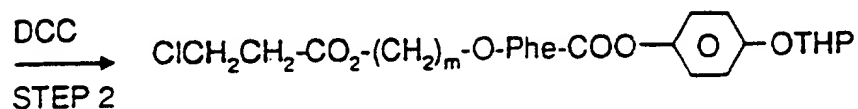
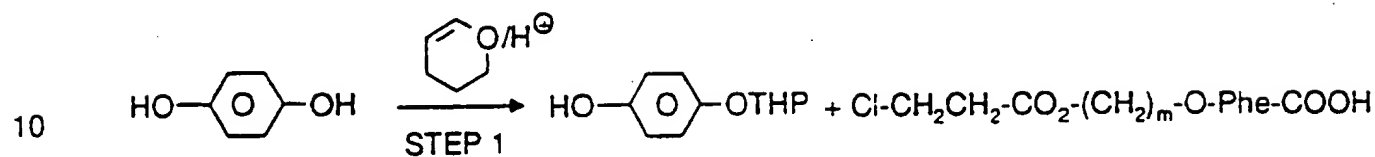
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Individual compounds of this type can be obtained according to schemes V to VII:

5 Scheme V

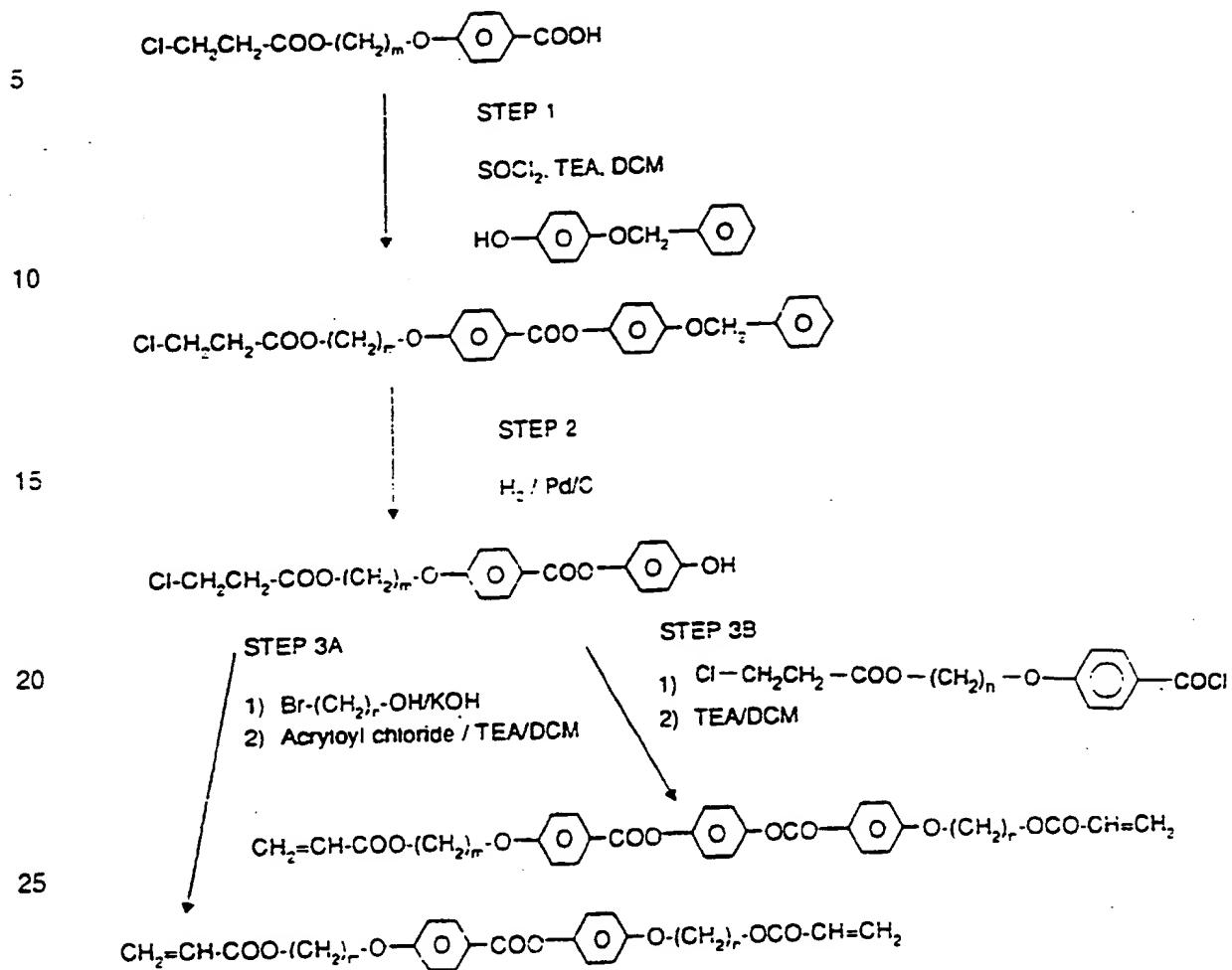


PTSA = p-toluenesulfonate

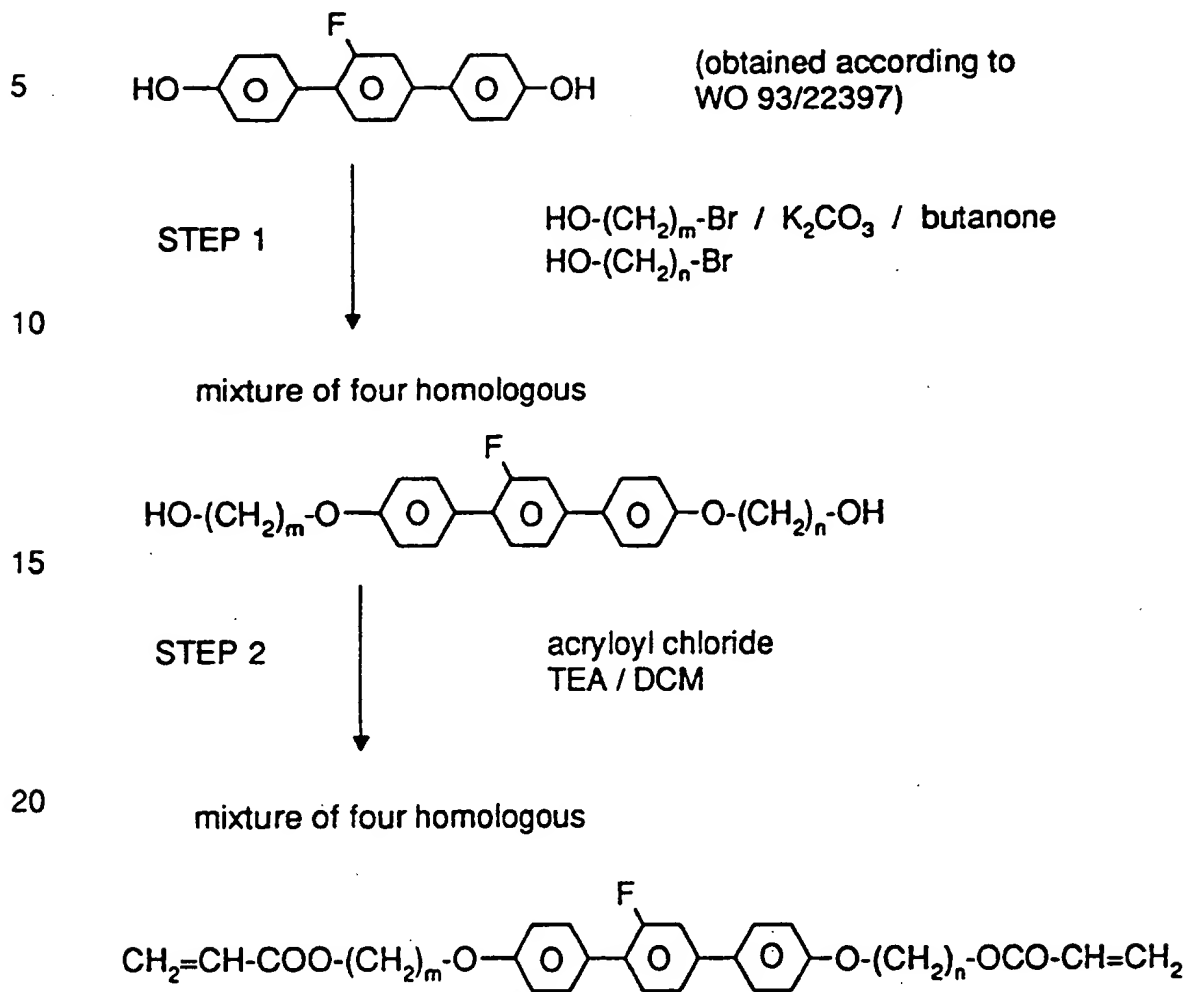
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Scheme VI



Scheme VII



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The reaction schemes mentioned above are to illustrate the invention without restricting it. The expert can choose other reaction methods without any inventive efforts.

5 In the following and in the preceding, all percentages given are percentages by weight. Temperatures are given in degrees Celsius.

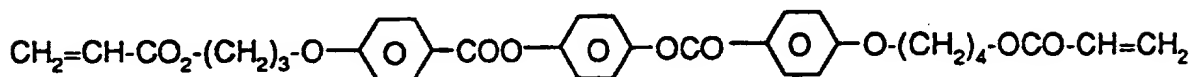
The following examples are intended to illustrate the invention without restricting it.

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Example 1

The reactive liquid crystalline compound (1)

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is prepared via the sequence of reaction steps shown in scheme V. In step 4 of scheme V 2.2 moles TFAA are added dropwise to a solution 2 moles of the phenol obtained in step 3 of scheme V and 2 moles of the benzoic acid in 2 l of DCM.

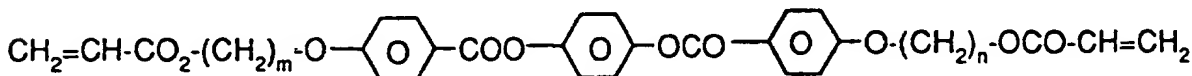
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The reaction mixture is stirred at room temperature for 16 hours. Then 5 moles of TEA in 1 l of DCM are added. The mixture is stirred for 16 hours. Aqueous work-up and column chromatography give (1) which shows K 111 S.

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The following compounds are obtained analogously:


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


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Compound No.	m	n	phase transition temperatures (°C)
(2)	3	5	K 76 N 160 I
(3)	4	5	K 67 N 165 I
(4)	4	6	K 78 S 82 N 159 I
(5)	5	6	K 78 S 80 N 162 I
Comp 1	4	4	K 105 N 164 I
Comp 2	5	5	K 91 N 167 I
Comp 3	6	6	K 105 (S 95) 152 I

### Example 2

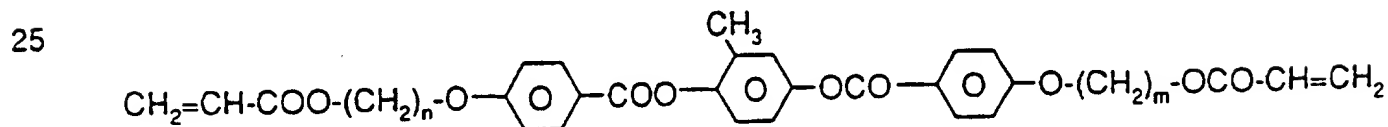
A mixture of 1 mol  $\text{Cl-CH}_2\text{CH}_2\text{-COO-(CH}_2)_4\text{-O-}$    $\text{-COOH}$ ,

1 mol  $\text{Cl-CH}_2\text{CH}_2\text{-COO-(CH}_2)_6\text{-O-}$    $\text{-COOH}$ , and 1 mol

p-hydroquinone is treated with 2.2 moles of TFAA followed by 2.5 moles of TEA.

A mixture consisting of 1 part comp 1 and 1 part comp 3 and 2 parts of Compound No. (4) is obtained which shows a melting point of 56 °C and a clearing point of 163 °C.

Analogously a mixture of the following compounds is obtained:



n	m	parts
3	3	1
6	6	1
3	6	1
6	3	1

35 This composition shows a melting point below 30 °C and a clearing point of 119 °C.

### Example 3

A mixture of 0.5 moles of biphenol, 1.3 moles of potassium hydroxide, 0.6 moles of 3-bromopropanol and 0.6 moles of 3 bromohexanol is heated in 2 litres of butanone for 16 hrs. After aqueous work-up a mixture of

1 part of  $\text{HO}-(\text{CH}_2)_3-\text{O}-\text{C}_6\text{H}_4-\text{C}_6\text{H}_4-\text{O}-(\text{CH}_2)_3-\text{OH}$

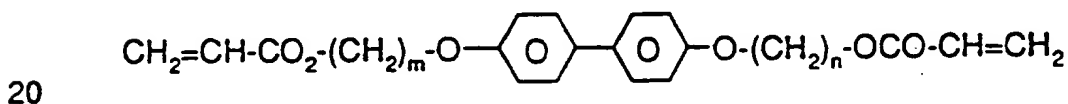
1 part of  $\text{HO}-(\text{CH}_2)_6-\text{O}-\text{C}_6\text{H}_4-\text{C}_6\text{H}_4-\text{O}-(\text{CH}_2)_6-\text{OH}$ , and

2 parts of  $\text{HO}-(\text{CH}_2)_6-\text{O}-\text{C}_6\text{H}_4-\text{C}_6\text{H}_4-\text{O}-(\text{CH}_2)_3-\text{OH}$

is obtained.

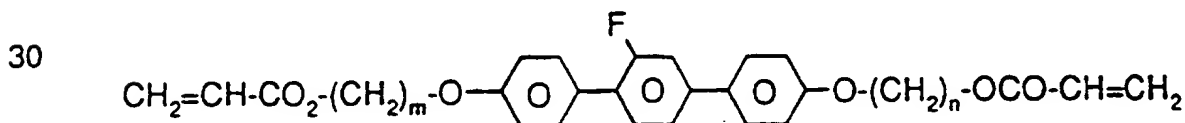
This mixture is treated with 1.2 moles of acryloyl chloride and 1.2 moles of TEA in 1.5 litres of dichloromethane DCM and refluxed for 3 hours.

After aqueous work-up the resulting reaction mixture is purified by column-chromatography to yield a mixture of diacrylates (6), (7) and (8).



Compound No.	m	n	parts
(6)	3	3	1
(7)	6	6	1
(8)	3	6	2

Analogously a mixture of compounds of formulae (9), (10), (11) and (12) is obtained





Compound No.		m	n	parts
5	(9)	3	3	1
	(10)	6	6	1
	(11)	3	6	1
	(12)	6	3	1

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### Claims

1. Direactive mesogenic compounds or mixtures thereof obtainable by

a) treating a mesogenic diol of formula I,



in which

MG is a mesogenic group,

with a mixture of the halides of formula II and III,

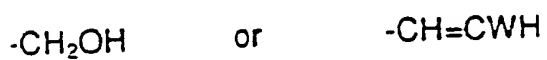


in which

$\text{X}^1$  and  $\text{X}^2$  are each independently Cl, Br or I

m and n are different integers between 1 and 20

$\text{R}^a$  and  $\text{R}^b$  are each independently groups selected from



wherein

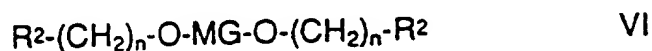
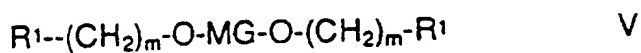
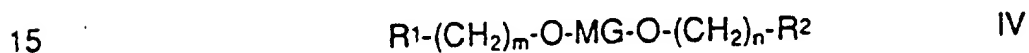
W is H,  $\text{CH}_3$  or Cl,

in the presence of a base, and

b) treating the resulting intermediate

- 5                    - in the case of  $R^a$  and  $R^b$  being  $-\text{CH}_2\text{OH}$ , with a vinyl derivative of formula  $\text{CH}_2=\text{CW}-(\text{CO})_a-\text{O}-$  or a reactive derivative thereof, in which  $a$  is 0 or 1
- 10                  - in the case of  $R^a$  and  $R^b$  being  $-\text{CH}=\text{CWH}$  with a perbenzoic acid.

2. Composition of direactive compounds comprising at least one compound of each formula IV, V and VI,

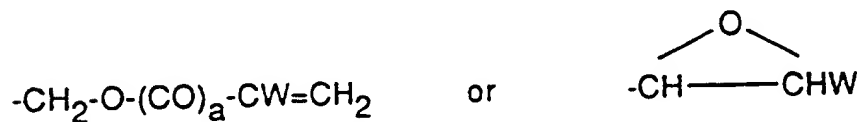


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in which MG,  $m$  and  $n$  have the meaning given, and

$\text{R}^1$  and  $\text{R}^2$  are each independently

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wherein  $a$  and  $W$  have the meaning given.

3. Direactive compound or mixture thereof according to claim 1 or 2

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in which

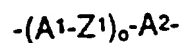
$m - n \geq 1.$

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4. Direactive compound or mixture thereof according to any of the preceding claim in which

MG is a mesogenic group of formula VII,

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VII

in which

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A<sup>1</sup> and A<sup>2</sup> are each independently

(a) 1,4-phenylene in which one or two CH groups may be replaced by N;

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(b) 1,4-cyclohexylene in which one or two non-adjacent CH<sub>2</sub> groups may be replaced by -O- or one -CH- group may be replaced by -C(CN)-;

(c) naphthalene-2,6-diyl;

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it being possible that group (a) is substituted by halogen cyano or alkyl with 1 to 6 C atoms,

Z<sup>1</sup>

is each independently -COO-, -O-CO-, -CH<sub>2</sub>-CH<sub>2</sub>-, -C≡C-, -CH<sub>2</sub>O-, -OCH<sub>2</sub>- or a single bond, and

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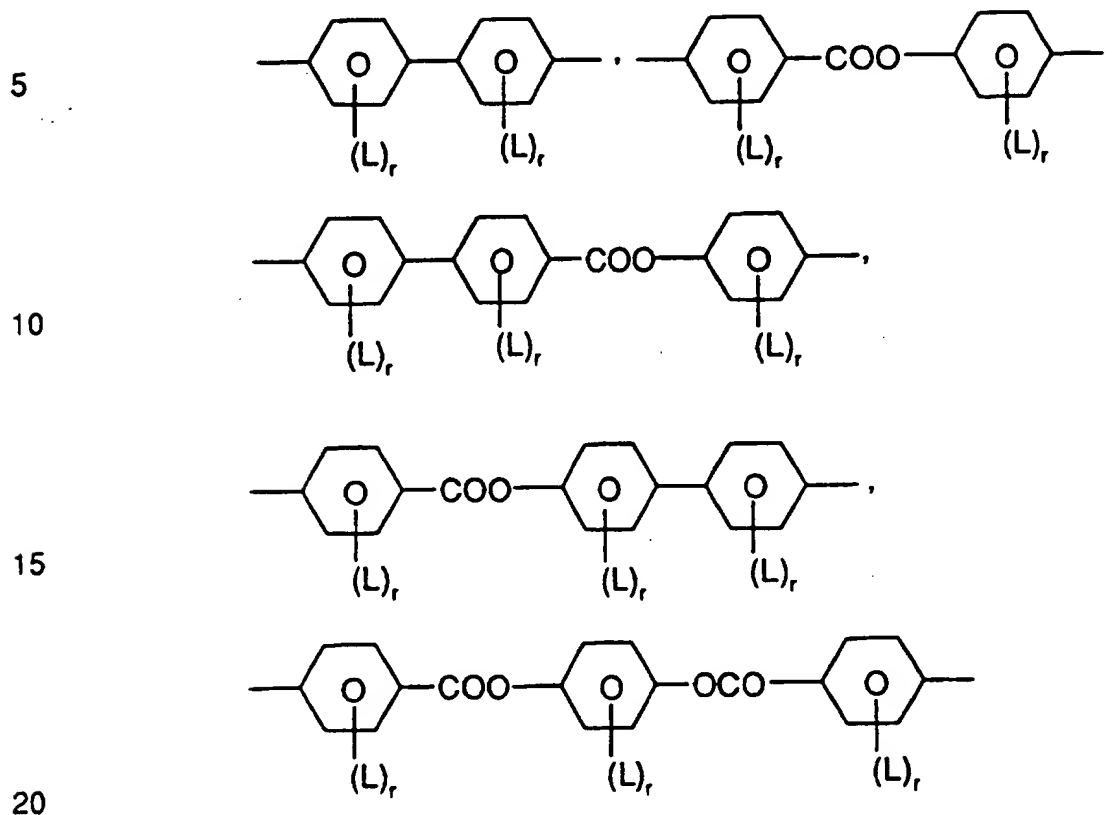
o

is 1, 2 or 3.

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5. Direactive compound or mixture thereof according to any of the preceding claims in which MG is selected from



in which

L is CH<sub>3</sub>, Cl, F, OCH<sub>3</sub> or -CO-CH<sub>3</sub>, and

r is 0, 1, 2 or 4.

6. Direactive compound or mixture thereof according to any of the preceding claims in which

n and m are given by the following table:

m	5	5	5	4	4
n	2	3	4	2	3

7. Direactive compound of formula IV A



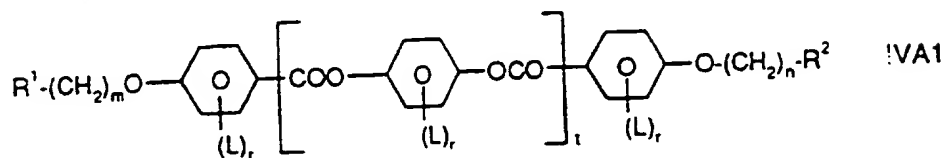
in which

$R^1$  and  $R^2$  have the meaning given,

$m$  and  $n$  are different integers between 2 and 10, and

$MG$  is a mesogenic group, the core of which being symmetrical.

8. Direactive compound according to claim 7 of the formula IVA1



in which

$R^1$ ,  $R^2$ ,

$L$  and  $r$  have the meaning given,

$m$  and  $n$  are different integers between 2 and 10, and

$t$  is 0 or 1.

9. Polymers prepared by polymerizing a monomer according to any of the preceding claims.

10. Chemical intermediate compounds or mixtures thereof useful in preparing direactive compounds or mixtures thereof according to any of Claims 1 to 8, comprising mesogen-containing molecules, said mesogens having two side chains attached thereto

which contain hydroxyl or vinyl group at the end thereof, said mesogens and said functional groups being separated by at least two to twenty spacer atoms, wherein both spacer groups have different chain length.

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Application No: GB 9502294.3  
Claims searched: 1-10

Examiner: Stephen Quick  
Date of search: 26 April 1996

**Patents Act 1977**  
**Search Report under Section 17**

**Databases searched:**

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.O): (not searched)

Int Cl (Ed.6): (not searched)

Other: Online: CAS ONLINE

**Documents considered to be relevant:**

Category	Identity of document and relevant passage	Relevant to claims
X	WO93/22397 A1 (MERCK PATENT), see compounds 4.1 (page 68; re claim 1) and 3.10 (page 67; re claim 10); acknowledged in this application	1 & 10 at least
X	Chem. Abs. 123:143045	1 & 10 at least
X	Chem. Abs. 122:33370	1 & 10 at least
X	Chem. Abs. 121:302740	1 & 10 at least
X	Chem. Abs. 121:256718	1 & 10 at least
X	Chem. Abs. 120:324302	1 & 10 at least
X	Chem. Abs. 120:272840	1 & 10 at least
X	Chem. Abs. 120:272839	1 & 10 at least
X	Chem. Abs. 120:165617	1 & 10 at least

X Document indicating lack of novelty or inventive step  
Y Document indicating lack of inventive step if combined with one or more other documents of same category.

A Document indicating technological background and/or state of the art.  
P Document published on or after the declared priority date but before the filing date of this invention.  
E Patent document published on or after, but with priority date earlier





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Application No: GB 9502294.3  
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Examiner: Stephen Quick  
Date of search: 26 April 1996

Category	Identity of document and relevant passage	Relevant to claims
X	Chem. Abs. 120:19350	1 & 10 at least
X	Chem. Abs. 119:227845	1 & 10 at least
X	Chem. Abs. 119:97676	1 & 10 at least
X	Chem. Abs. 118:30690	1 & 10 at least
X	Chem. Abs. 116:42907	1 & 10 at least
X	Chem. Abs. 115:115251	1 & 10 at least
X	Chem. Abs. 113:192043	1 & 10 at least
X	Chem. Abs. 113:79530	1 & 10 at least

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E Patent document published on or after, but with priority date earlier

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